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L6 ANSWER 1 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2004:947819 CAPLUS <<LOGINID::20080310>>

DOCUMENT NUMBER: 142:36754

TITLE: The X-Ray Structure of RANTES: Heparin-Derived

Disaccharides Allows the Rational Design of Chemokine

Inhibitors

AUTHOR(S): Shaw, Jeffrey P.; Johnson, Zoe; Borlat, Frederic;

Zwahlen, Catherine; Kungl, Andreas; Roulin, Karen; Harrenga, Axel; Wells, Timothy N. C.; Proudfoot,

Amanda E. I.

CORPORATE SOURCE: Serono Pharmaceutical Research Institute, Geneva,

1228, Switz.

SOURCE: Structure (Cambridge, MA, United States) (2004

), 12(11), 2081-2093

CODEN: STRUE6; ISSN: 0969-2126

PUBLISHER: Cell Press
DOCUMENT TYPE: Journal
LANGUAGE: English

AΒ The biol. activity of chemokines requires interactions with cell surface proteoglycans. We have determined the structure of the chemokine RANTES (regulated on activation normal T cell expressed) in the presence of heparin-derived disaccharide analogs by x-ray crystallog. These structures confirm the essential role of the BBXB motif in the interaction between the chemokine and the disaccharide. Unexpected interactions were observed in the 30s loop and at the amino terminus. Mutant RANTES mols. were designed to abrogate these interactions and their biol. activity examined in vivo. The K45E mutant within the BBXB motif lost the capacity to bind heparin and the ability to elicit cellular recruitment. The Y3A mutant maintained its capacity to bind heparin but was unable to elicit cellular recruitment. Finally, a tetrasaccharide is the smallest oligosaccharide which effectively abolishes the ability of RANTES to recruit cells in vivo. These crystallog. structures provide a description of the mol. interaction of a chemokine with glycosaminoglycans.

IT 145882-46-8D, RANTES complex

RL: BSU (Biological study, unclassified); PRP (Properties); BIOL (Biological study)

(crystal structure of RANTES:heparin-derived disaccharides)

RN 145882-46-8 CAPLUS

CN  $\alpha$ -D-Glucopyranose, 2-deoxy-4-O-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)- (9CI) (CA INDEX NAME)

REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 2 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2004:270131 CAPLUS <<LOGINID::20080310>>

DOCUMENT NUMBER: 140:287660

Preparation and HPLC of oligosaccharides obtained by TITLE:

heparinase-catalyzed depolymerization of low molecular

weight of heparins

INVENTOR(S): Mourier, Pierre; Viskov, Christian

PATENT ASSIGNEE(S): Aventis Pharma S.A., Fr. SOURCE: PCT Int. Appl., 23 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT: 3

PATENT INFORMATION:

PA:	PATENT NO.					KIND DATE			APPLICATION NO.					DATE				
	WO 2004027087 WO 2004027087								WO 2003-FR2782					20030922 <				
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CA	CA 2499537			A1		2004	0401	(	CA 2	2003-	2499	537		20030922 <				
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BR	BR 2003014654							BR 2003-14654					20030922 <					
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A method for heparinase-catalyzed depolymn. of low mol. weight of heparins and HPLC structure determination of the corresponding oligosaccharides and reduced

oligosaccharides are reported.

674789-50-5P 674789-51-6P ΙT

> RL: BPN (Biosynthetic preparation); PRP (Properties); RCT (Reactant); BIOL (Biological study); PREP (Preparation); RACT (Reactant or reagent)

(preparation and HPLC of oligosaccharides obtained by heparinase-catalyzed depolymn. of low mol. weight of heparins)

RN 674789-50-5 CAPLUS CN  $\beta$ -D-Glucopyranose, 2-deoxy-4-O-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)-, disodium salt (9CI) (CA INDEX NAME)

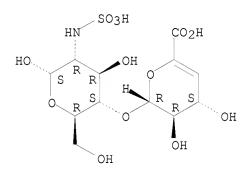
Absolute stereochemistry.

●2 Na

RN 674789-51-6 CAPLUS

CN  $\alpha$ -D-Glucopyranose, 2-deoxy-4-O-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)-, disodium salt (9CI) (CA INDEX NAME)

Absolute stereochemistry.



●2 Na

L6 ANSWER 3 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2004:249315 CAPLUS <<LOGINID::20080310>>

DOCUMENT NUMBER: 140:287659

TITLE: Preparation and HPLC of oligosaccharides obtained by

heparinase-catalyzed depolymerization of low molecular

weight of heparins

INVENTOR(S): Mourier, Pierre; Viskov, Christian

PATENT ASSIGNEE(S): Aventis Pharma S. A., Fr. SOURCE: Fr. Demande, 23 pp.

CODEN: FRXXBL

DOCUMENT TYPE: Patent LANGUAGE: French

PA:	PATENT NO.				KIND DATE		APPLICATION NO.					DATE						
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										CN 2	003-	8225	62		A3 20	00309	922	
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AB A method for heparinase-catalyzed depolymn. of low mol. weight of heparins and HPLC structure determination of the corresponding oligosaccharides and reduced

oligosaccharides are reported.

IT 674789-50-5P 674789-51-6P

RL: BPN (Biosynthetic preparation); PRP (Properties); RCT (Reactant); BIOL (Biological study); PREP (Preparation); RACT (Reactant or reagent)

(preparation, hydride-reduction, and HPLC of oligosaccharides obtained by heparinase-catalyzed depolymn. of low mol. weight of heparins)

RN 674789-50-5 CAPLUS

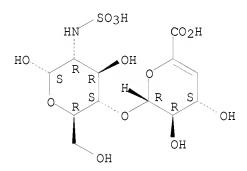
CN  $\beta$ -D-Glucopyranose, 2-deoxy-4-O-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosy1)-2-(sulfoamino)-, disodium salt (9CI) (CA INDEX NAME)

●2 Na

RN 674789-51-6 CAPLUS

CN  $\alpha$ -D-Glucopyranose, 2-deoxy-4-O-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)-, disodium salt (9CI) (CA INDEX NAME)

Absolute stereochemistry.



•2 Na

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 4 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1999:558151 CAPLUS <<LOGINID::20080310>>

DOCUMENT NUMBER: 131:334237

TITLE: Disaccharide Analysis and Molecular Mass Determination

to Microgram Level of Single Sulfated

Glycosaminoglycan Species in Mixtures Following

Agarose-Gel Electrophoresis

AUTHOR(S): Volpi, Nicola

CORPORATE SOURCE: Department of "Biologia Animale,", Biological

Chemistry Section, University of Modena, Modena, Italy

SOURCE: Analytical Biochemistry (1999), 273(2),

229-239

CODEN: ANBCA2; ISSN: 0003-2697

PUBLISHER: Academic Press

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The separation of sulfated glycosaminoglycans in mixts. by agarose-gel electrophoresis and the recovery of single polysaccharide bands has been applied to the characterization of polysaccharides extracted from tissues without previous purification of single species. Sulfated glycosaminoglycans, heparin with its two components, slow-moving and fast-moving, heparan sulfate, dermatan sulfate, and chondroitin sulfate, were separated to microgram level by conventional agarose-gel electrophoresis. After their separation, they were fixed in the agarose-gel matrix by precipitation in a cetyltrimethylammonium bromide solution, making them visible on a dark background. After recovery of gel containing the fixed bands, high temps. (90° for 15 min) were necessary to dissolve the gel matrix, and a solution of NaCl (3 M) was used to release sulfated polysaccharides from the complex with cetyltrimethylammonium. After precipitation of glycosaminoglycans in

the presence of ethanol, the recovery of slow-moving heparin, fast-moving heparin, heparan sulfate, dermatan sulfate, and chondroitin sulfate was from 1 to 10  $\mu g$ , with a percentage greater than 45% and a purity above 90%. Sulfated glycosaminoglycans in mixts, recovered from gel matrix as single species were evaluated for purity and characterized for unsatd, disaccharides after treatment with bacterial lyases (heparinases for heparin and heparan sulfate samples, and chondroitinases for dermatan sulfate and chondroitin sulfate) and mol. mass. Bovine lung and heart glycosaminoglycans were extracted and separated into single species by agarose-gel

electrophoresis and recovered from gel matrix after treatment in cetyltrimethylammonium solution Unsatd. disaccharides pattern, the sulfate to carboxyl ratio, and the mol. mass of each single polysaccharide species were determined (c) 1999 Academic Press.

IT <u>145882-46-8</u>

RL: ANT (Analyte); BOC (Biological occurrence); BSU (Biological study, unclassified); ANST (Analytical study); BIOL (Biological study); OCCU (Occurrence)

(disaccharide anal. and mol. mass determination to microgram level of single sulfated glycosaminoglycan species in mixts. following agarose-gel electrophoresis)

RN 145882-46-8 CAPLUS

CN  $\alpha$ -D-Glucopyranose, 2-deoxy-4-O-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)- (9CI) (CA INDEX NAME)

L6 ANSWER 5 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1994:293407 CAPLUS <<LOGINID::20080310>>

DOCUMENT NUMBER: 120:293407

TITLE: Fractionation of heparin, dermatan sulfate, and

chondroitin sulfate by sequential precipitation: a method to purify a single glycosaminoglycan species

from a mixture Volpi, Nicola

CORPORATE SOURCE: Dep. "Biol. Anim.", Univ. Modena, Modena, 41100, Italy

SOURCE: Analytical Biochemistry (1994), 218(2),

382-91

CODEN: ANBCA2; ISSN: 0003-2697

DOCUMENT TYPE: Journal LANGUAGE: English

AB Purified heparin, dermatan sulfate, and chondroitin sulfate in mixts. were fractionated by sequential precipitation with increasing vols. of acetone and analyzed by agarose-gel electrophoresis and for Mr, charge d., constituent disaccharides, and anticoagulant activity (for heparin). Purified glycosaminoglycans are generally utilized for pharmaceutical purposes and show physicochem. properties of glycosaminoglycans used as drugs. Heparin is the first glycosaminoglycan to precipitate at low percentages of acetone.

The

AUTHOR(S):

relative amount of slow moving and fast moving components, the Mr and charge d., and the disaccharide pattern of fractionated heparin depend on the percentage of solvent. The activated partial thromboplastin time activity of fractions composed of heparin decreases with the charge d. and Mr. Dermatan sulfate is precipitated by acetone over a narrow range (0.6-0.7 volume,

37-41%), and one of these fractions is constituted by 100% of this polysaccharide. These species of dermatan sulfate have different percentages of constituent disaccharides compared to the native polysaccharide. Nonsulfated disaccharide and disaccharide-6-sulfate are enriched. The dermatan sulfate species precipitated by acetone are also enriched

in disaccharide-4,6-disulfate. Chondroitin sulfate is the most soluble glycosaminoglycan in mixed acetone/water solvent. It begins to precipitate at 0.8 vol (44%) of acetone. Different species of chondroitin sulfate can be recovered by precipitation at different percentages of solvent, and they show a decrease in Mr and charge d. depending on the percentage of acetone. The chondroitin sulfate species fractionated are also enriched in disulfated disaccharides compared to native polysaccharide. A different distribution of the three disulfated disaccharides can be pointed out in the fractionated chondroitin sulfate. Sequential precipitation performed by carefully

increasing acetone percentages can help obtain purified species of glycosaminoglycan with desired properties from a mixture and tissue exts., and achieve savings in time.

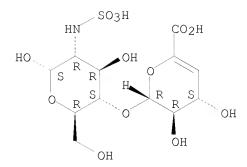
IT 145882-46-8

RL: ANST (Analytical study)

(of heparin, after acetone precipitation)

RN 145882-46-8 CAPLUS

CN  $\alpha$ -D-Glucopyranose, 2-deoxy-4-0-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)- (9CI) (CA INDEX NAME)



ANSWER 6 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

1994:49369 CAPLUS <<LOGINID::20080310>> ACCESSION NUMBER:

DOCUMENT NUMBER: 120:49369

TITLE: "Fast moving" and "slow moving" heparins, dermatan

sulfate, and chondroitin sulfate: qualitative and quantitative analysis by agarose-gel electrophoresis

AUTHOR(S): Volpi, Nicola

Dep. "Biol. Anim.", Univ. Modena, Modena, Italy CORPORATE SOURCE:

Carbohydrate Research (1993), 247, 263-78 SOURCE:

CODEN: CRBRAT; ISSN:  $00\overline{08-6215}$ 

DOCUMENT TYPE: Journal LANGUAGE: English

Heparin from beef intestinal mucosa, dermatan sulfate from beef intestinal AΒ mucosa, and chondroitin sulfate from bovine trachea were extracted and purified, and their structures and physico-chemical properties were evaluated by different techniques (disaccharide patterns by specific enzymic cleavage, relative mol. mass by high-performance size-exclusion chromatog., sulfate-to-carboxyl ratio by potentiometric determination).

was fractionated into "slow moving" and "fast moving" fractions by selective precipitation as the barium salt at different temps. The "fast moving"

and "slow moving" components of heparin, dermatan sulfate, and chondroitin sulfate were utilized to run calibration curves in agarose-gel electrophoresis. Mixts. containing different amts. of these glycosaminoglycans were made and separated by agarose-gel electrophoresis, and these were analyzed quant. For anal. of relative amts., the area of each individual component of mixts., obtained by photodensitometric readings, was divided by the sum of the areas of all glycosaminoglycans and expressed as a percentage. For anal. of absolute amts., the area under the curve for each component of mixts. was fitted to specific calibration curves, and the amount of each glycosaminoglycan was calculated in ug. quant. procedure performed by analyzing absolute amts. was used to obtain an accurate quant. evaluation of each component in mixts. of glycosaminoglycans utilized for pharmaceutical purposes. A sensitive method was developed for the evaluation of very small amts. (0.2%

weight/weight)

of possible glycosaminoglycans as contaminants in prepns. of a single species of glycosaminoglycan. This technique requires specific enzymic degradation by bacterial lyases, separation in agarose-gel electrophoresis, and quant. anal. by photodensitometric anal. and specific calibration curves.

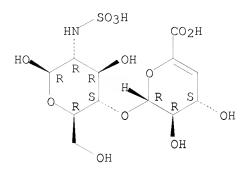
ΤТ 151505-06-5

Heparin

RL: ANST (Analytical study) (release and anal. of, of heparins) RN 151505-06-5 CAPLUS

CN  $\beta$ -D-Glucopyranose, 2-deoxy-4-0-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L6 ANSWER 7 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1994:8885 CAPLUS <<LOGINID::20080310>>

DOCUMENT NUMBER: 120:8885

TITLE: Conformation of the unsaturated uronic acid residues

of glycosaminoglycan disaccharides

AUTHOR(S): Ragazzi, M.; Ferro, D. R.; Provasoli, A.; Pumilia, P.;

Cassinari, A.; Torri, G.; Guerrini, M.; Casu, B.;

Nader, H. B.; Dietrich, C. P.

CORPORATE SOURCE: Ist. Chim. Macromol., CNR, Milan, I-20133, Italy

SOURCE: Journal of Carbohydrate Chemistry (1993),

12(4-5), 523-35

CODEN: JCACDM; ISSN: 0732-8303

DOCUMENT TYPE: Journal LANGUAGE: English

Ι

GΙ

AB Mol. mechanics calcns. (using the REFINE package) have been performed on a series of disaccharides, e.g. I (R = Ac, SO3H), obtained by cleavage of glycosaminoglycans with lyases, in order to examine the effect of chemical environment on the conformation of the 4,5-unsatd. uronic acid residue. The disaccharides were derived from heparin and heparan sulfate, hyaluronic acid, chondroitin, chondroitin-4-sulfate, and chondroitin-6-sulfate.

IT 145882-46-8 151505-06-5

RL: PRP (Properties)

(conformation and mol. mechanics of, NMR in relation to)

RN 145882-46-8 CAPLUS

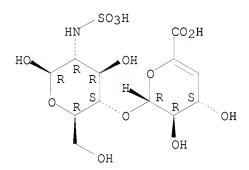
CN  $\alpha$ -D-Glucopyranose, 2-deoxy-4-0-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

RN 151505-06-5 CAPLUS

CN  $\beta$ -D-Glucopyranose, 2-deoxy-4-0-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L6 ANSWER 8 OF 8 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1993:96610 CAPLUS <<LOGINID::20080310>>

DOCUMENT NUMBER: 118:96610

TITLE: One- and two-dimensional proton NMR characterization

of two series of sulfated disaccharides prepared from chondroitin sulfate and heparan sulfate/heparin by

bacterial eliminase digestion

AUTHOR(S): Yamada, Shuhei; Yoshida, Keiichi; Sugiura, Makiko;

Sugahara, Kazuyuki

CORPORATE SOURCE: Dep. Physiol. Chem., Kobe Women's Coll. Pharm., Kobe,

658, Japan

SOURCE: Journal of Biochemistry (Tokyo, Japan) (1992

), 112(4), 440-7

CODEN: JOBIAO; ISSN: 0021-924X

DOCUMENT TYPE: Journal LANGUAGE: English

AB The 1H-NMR spectra of eight unsatd. disaccharides obtained by bacterial

eliminase digestion of chondroitin sulfate and of heparan sulfate/heparin were recorded in order to construct an NMR data base of sulfated oligosaccharides and to investigate the effects of sulfation on the proton chemical shifts. These shifts were assigned by two-dimensional HOHAHA (homonuclear Hartmann-Hahn) and COSY (correlation spectroscopy) methods. The results indicated the following. Two sets of proton signals were observed, corresponding to the  $\alpha$  and  $\beta$  anomers of these disaccharides, except those containing N-sulfated GlcN (2-deoxy-2-amino-Dqlucose), in which only one set of signals appeared, corresponding to the  $\alpha$  anomer. Signals of protons bound to an O-sulfated carbon atom and those bound to the immediately neighboring carbon atoms were shifted downfield by 0.4-0.7 and 0.07-0.3 ppm, resp. For the disaccharides containing the N-sulfated GlcN, the signals of the protons bound to C-2 and C-3 were shifted upfield by 0.6 and 0.15 ppm, resp., but that of C-1 was shifted downfield by 0.25 ppm when compared with those of the corresponding N-acetylated disaccharides. For the chondroitin sulfate disaccharides sulfated on the C-4 position of GalNAc (2-deoxy-2-N-acetylamino-Dgalactose) or the C-2 position of  $\Delta GlcA$  (D-gluco-4-enepyranosyluronic acid), the signal of the H-3 proton of  $\Delta GlcA$  or the H-4 proton of GalNAc was shifted upfield by 0.1-0.15 ppm, indicating the steric interaction of the two sugar components. These effects of sulfation on chemical shifts are additive.

IT <u>145882-46-8</u>

RL: BIOL (Biological study)

(of heparan sulfate and heparin, structure of, NMR study of)

RN 145882-46-8 CAPLUS

CN  $\alpha$ -D-Glucopyranose, 2-deoxy-4-0-(4-deoxy- $\alpha$ -L-threo-hex-4-enopyranuronosyl)-2-(sulfoamino)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

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L5

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FILE 'REGISTRY' ENTERED AT 16:35:24 ON 10 MAR 2008 STRUCTURE UPLOADED

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L6 8 S L5 AND PY<=2005 L7 0 S L6 AND (HGF OR HEPATOCYTE)

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L1 HAS NO ANSWERS
L1 STR

Structure attributes must be viewed using STN Express query preparation.

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FULL ESTIMATED COST	ENTRY 52.36	SESSION 231.85
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE ENTRY	TOTAL SESSION
CA SUBSCRIBER PRICE	-6.40	-6.40

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STN INTERNATIONAL SESSION SUSPENDED AT 16:39:49 ON 10 MAR 2008